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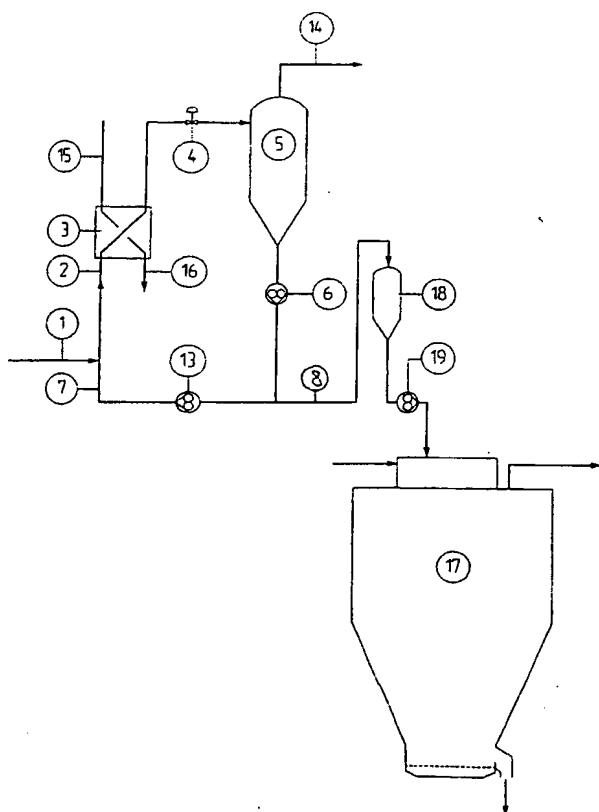
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[Continued on next page]

- (54) Title:** A METHOD AND A SYSTEM FOR CONVERTING LIQUID PRODUCTS INTO FREE-FLOWING POWDERS WITH PRE-COOLING



(57) Abstract: The present invention relates to a method and a system for converting liquid products for the cheese making or casein producing industry into substantially free-flowing powdery products, by subjecting the liquid product to heating to a temperature above the crystallisation temperature of any component in the liquid product in a heat exchanger, flash separating volatile components from said heated liquid product to obtain a past concentrate, pre-cooling a fraction of said past concentrate, and drying said combination product. By the pre-cooling it is possible to create lactose crystals by an extremely rapid in-line pre-cooling without any significant increase in viscosity (which would lead to an un-pumpable paste). It is assumed that only a fraction of the paste gets into contact with the walls of the cooler causing a rapid formation of a high number of seed crystals. Further, it is assumed that the seed promote the formation of lactose crystals during the subsequent drying process.

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A method and a system for converting liquid products into free-flowing powders with pre-cooling

Field of the invention

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The present invention relates to a method and a system for converting liquid products from the cheese making or casein producing industry into substantially free-flowing powdery products.

Background of the invention

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The traditional cheese making and casein manufacturing industry produce a series of liquid products and by-products, in the present context termed whey: acid whey, sweet whey, salt whey, permeates from the production of whey protein or milk protein concentrates. Other liquid products are lactose solution, mother liquor from lactose crystallisation, demineralised whey and the like.

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Depending on origin the whey contains 5.5-6.5 % total solids, wherein 0.7-0.9 % protein, 0.05 % fat, 4.2-4.6 % lactose and 0.5-0.8 % ash.

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Usually, after a number of pre-treatments, the aqueous whey is a solution in which the lactose is in an equilibrium of 60 % beta- and 40 % alpha molecular form. This mixture is very hygroscopic and unsuitable for drying for creating a free flowing, non-caking powder.

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However, the whey solution may be dried into a suitable powder if a major part of the lactose during the processing is crystallised into the alpha monohydrate form.

Description of prior art

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In the prior art several processes are known for converting liquid products, such as whey as defined above into substantially free-flowing, non-caking powdery products. Major processes are mentioned in PCT application WO 00/72692 (APV Nordic, Anhydro) to which reference is made.

5 In processes commonly carried out by industry, the whey solution is concentrated up till 50 or 66 % total solids and cooled slowly batchwise in large tanks for crystallisation of lactose. After crystallisation, the concentrate is spray dried to its final moisture content and cooled in a fluid bed (Masters, Spray Drying Handbook, 4 ed. 1985, p. 596)

10 In WO 00/72692 mentioned above, a process is described in which the liquid is concentrated to at least 65 % TS or at least 70 % by flash evaporation while keeping the temperature above the crystallisation temperature of the liquid product. Crystallisation is then carried out in one or more steps by cooling, and the product is finally dried in a Spin Flash®).

15 It has been found, however, that the crystallisation process makes the whey paste from the evaporator highly viscous and difficult to handle and disintegrate in the Spin Flash dryer when conducted in large scale production. The process requires relatively heavy duty mixing and feeding equipment.

20 Therefore, there is an unfulfilled demand for a process for converting liquid products, such as whey, into a substantially free-flowing powdery product, and which process makes less demand on mixing equipment.

Summary of the invention

25 The present invention relates to a method for converting a liquid product into substantially free-flowing powder, said method comprising the steps of

- heating the liquid product to a temperature above the crystallisation temperature of any component in the liquid product in a heat exchanger,
- flash separating volatile components from said heated liquid product to obtain a
- 30 paste concentrate,
- pre-cooling a fraction of said paste concentrate, and
- drying said combination product.

35 Thus, by the present invention a combination product of cooled paste concentrate and uncooled paste concentrate is obtained in a cooler before drying.

By the present invention it has been possible to concentrate the liquid product to a very high concentration of solids, and pre-cooling while maintaining the pumpable nature of the paste.

5 Furthermore, the invention relates to a method for converting liquid whey products into substantially free-flowing powder comprising the step of concentration and pre-cooling as defined above and drying, obtaining free-flowing powder.

In a preferred embodiment, a free-flowing, non-caking powder is obtained.

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Accordingly, the present invention further relates to a system for concentrating a liquid product, having a unit comprising means (2) for feeding a heat exchanger, a heat exchanger (3) and a separator vessel (5) and means for pre-cooling (18) a fraction of said concentrate.

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Further, the invention relates to a system for converting whey into a powdery product comprising means for concentrating a liquid product to obtain a pre-cooled concentrate as defined above, and means for air drying said pre-cooled concentrate to obtain free-flowing, non-caking powder, which means are interconnected by a main conduit.

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Drawing

The drawing is a schematic diagram of a system according to the invention for carrying out the method.

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Detailed description of the method

When concentrating liquid products as defined above, it is of importance to control crystallisation, especially of lactose. Lactose crystals inevitably form deposits on heat exchanger surfaces and prevent heat transfer. Therefore, the temperature of the liquid must be maintained above the temperature at which crystals form during the evaporation process. In particular, no formation of α -lactose crystals occurs when the liquid product is heated to the temperature above crystallisation temperature. High temperatures on the other hand promote the undesired browning (Mail-

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lard) reaction and denaturation of proteins (Reyes, F.G.G. (1982) J. Food Sci., 47, 1376-7). The extent of these undesired reactions is minimised by a short residence time at elevated temperatures in the heater. Therefore, a heat exchanger, with a short contact time, is preferred. By the term heat exchanger is meant any suitable heater providing a fast and even heating. Thus a heat exchanger may be any conventional heat exchanger, such as a plate heat exchanger, a "scraped-surface heat exchanger" and a tube heat exchanger. A plate heat exchanger is preferred, such as a heat exchanger having a high ratio of height to width, such as a ratio from 2.5 to 3.5.

The heat exchanger may also be an electroheating means providing volumetric heating by means of direct electrical resistance heating, also called ohmic heating. Thus, the liquid whey product is preferably heated to at least 80°C in a heat exchanger. By heating to at least 80°C, more preferred to at least 85°C, even more preferred to at least 90°C, the risk of crystallisation in the heat exchanger is significantly reduced.

Boiling induces deposits in the exchanger; it is suppressed by a pressure control valve (4).

After heating, the product is subjected to a flash evaporation process. Thus, after being heated in the heat exchanger, the liquid product is transferred to the separator vessel by means of for example a connecting line between the heat exchanger and the separator vessel, to be flash separated in the separator. The principle of flash evaporation according to this invention is well known, see e.g. Perry's Chemical Engineer' Handbook, 6 Ed. (1984), pp.11-35, 11-40. A flow of liquid is heated in a heat exchanger and allowed to expand into a vessel where the actual solvent (volatile components) will evaporate with a corresponding drop of temperature in the liquid phase. It is understood that the temperature rise in the heat exchanger and the temperature drop of the liquid in the flash are substantially equal.

The volatile components will be any components in the liquid product capable of evaporation at the temperature and pressure of the separator vessel. During flash separation the volatile components of the liquid products are separated from the liquid product, whereby a liquid concentrate of the liquid product is obtained. After

separation the formed vapour is removed by means of a condenser or vacuum pump.

5 The temperature of the separator vessel is equal to or a few degrees below the temperature of the heated liquid product. Accordingly, the temperature of the liquid product in the separator vessel is preferably in the range of 65-96°C, more preferably in the range of 70-96°C, even more preferably in the range of 80-96°C.

10 Furthermore, the pressure in the vessel wherein the flash separation is carried out is preferably regulated to a pressure below the vapour pressure of the heated liquid product. Thus, the separation is preferably conducted under a pressure in the range of 0.4-1.2 bar. More preferred a vacuum is applied, such as 0.5-0.9 bar.

15 The concentration method is preferably applied to whey products, whereby it is desired to obtain as high a total solids (TS) concentration as possible. In a preferred embodiment, concentration of raw whey leads to at least 75% TS at 75°C whereas concentration of permeate leads to at least 82% TS at a temperature of about 96°C. In the present context, the term "TS" means total solids in a product or an intermediate as kg dry solid/kg product, as measured by weight loss in an oven at 102°C
20 for 4 hours.

If such products are cooled a few degrees, a very high number of small lactose crystals will form immediately.

25 However, according to the method of the present invention, it has been found that a rapid cooling and subsequent formation of crystals does not render the paste unpumpable. So, according to the present invention, the pre-cooled product is pumped to the atomizer of a conventional spray dryer with integrated fluid bed, see e.g. Masters, K., Spray Drying Handbook, 5 Ed., (1991), p. 609.

30 By the present method it has become possible to concentrate and dry liquid products, in particular liquid whey products, and obtain a substantially free-flowing, non-caking powder by a process hitherto unknown to industry.

5 The powder flowability may be measured according to Cheremisinoff (see below) whereby a free-flowing powder is having an angle of repose of 30 or less. The caking properties are estimated by examining the crust formed on a spoonful of powder, subjected to 60 % relative humidity at 20°C for 24 hours. A non-caking powder does not form any crust under these conditions. A non-caking powder is more stable than caking powders.

10 When the concentration process is carried out according to the invention a powder having very good properties in relation to flowability and preferably also cakability is obtained.

15 By the term a powdery product is meant a product normally considered as powder, i.e. having a particle size corresponding to powder, such a preferably above 0.1 μm and preferably below 2 mm. The particle size is measured as the approximate diameter of the particle.

20 The formation of crystals during a time and space requiring process upstream of the drying is a characteristic feature of prior art processes for converting liquid whey products into free-flowing, non-caking powders.

25 The pre-cooling is preferably conducted in a cooler having a cooling jacket. The passing rate of the paste concentrate through the cooler is adjusted to the filling degree of the cooler as well as the other parameters of the system. In a preferred embodiment at most 25 % of the paste concentrate is cooled, such as preferably at most 20 % of the paste concentrate. In particular the cooler cannot be a plate cooler, since the whey would deposit on the plates during cooling leading to smaller yield as well as down periods for the system.

30 By the present method it has been found that it is possible to create lactose crystals by an extremely rapid in-line pre-cooling without any significant increase in viscosity (which would lead to an un-pumpable paste). It is assumed that only a fraction of the paste gets into contact with the walls of the cooler causing a rapid formation of a high number of seed crystals. Further, it is assumed that the seed promote the formation of lactose crystals during the subsequent drying process.

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This means that there is no need for crystallisation tanks or for heavy-duty mixers. Preferably at most 50 % of the paste concentrate is crystallised during cooling, such as more preferably at most 25 % of the paste concentrate.

5 The paste concentrate obtained may be transferred directly to the pre-cooling step and subsequently dried in a dryer. However, it has been found that the concentration process is further improved if it comprises recirculation of at least a part of the concentrate. The recycled concentrate is then mixed with the liquid product before being fed to the heat exchanger.

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The recirculation is preferably carried out by continuous recirculation of a predetermined partial flow of the liquid concentrate to the heat exchanger. Preferably, at least 75 % of the liquid concentrate is continuously recycled to be mixed with liquid product and then entered into the heat exchanger to be reheated and subjected to a further separating step. In an even more preferred embodiment at least 90 % of the liquid concentrate is continuously recycled. This may be accomplished by using conduits having different diameters when transferring the liquid product and the liquid concentrate to the heat exchanger. In particular a ratio of the cross-section area of the liquid concentrate conduit to the cross-section area of the liquid product conduit is at least 10:1, such as at least 25:1, more preferred at least 30:1, yet more preferred at least 50:1.

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The ratio of liquid concentrate to liquid product in the mixture feeding the heat exchanger is preferably at least 3:1, such as at least 10:1, more preferred at least 25:1, most preferred at least 50:1.

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Furthermore, the high percentage of recycling also leads to a high content of solids in the mixture of recycled liquid concentrate and liquid product. The high content of solids reduces the water activity and increases the viscosity leading to a low migration velocity of molecules. Without being bound by theory, it is believed that due to a high solid content in the liquid product the lactose molecules are "masking" the protein molecules, inhibiting unfolding and denaturation of the protein.

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Highly concentrated whey is very viscous. However, due to the visco-elastic and thixotropic nature of whey concentrate, the circulation rate has a great influence on

the viscosity of the liquid concentrate. The circulation rate is controlled to any suitable rate with respect to the apparatus in use. The rate is preferably as high as possible reducing the viscosity of the liquid concentrate. As a measure of the preferred flow rates in the separating vessel and the conduit through positive displacement pump a preferred ratio between their diameters is indicated. The diameter of the flash separating vessel is preferably in the range of from 3 to 30 times the diameter of the conduit leading to the pump, more preferably the ratio of the diameter of the separating vessel to the diameter of the conduit is in the range from 10:1 to 20:1.

Various pretreatment of the liquid product before entering the line leading to the heat exchanger may be carried out. Also, a pre-concentration step may be conducted. In particular with respect to whey a pre-concentration may be conducted whereby the solid content is increased from the starting concentration of about 6 % to about 58 %. The pre-concentration may be carried out by any suitable method known to the skilled person.

The method has been described with respect to one concentration unit comprising one heat exchanger and one flash separator. However, the method may also be conducted in a system comprising two or more units. Thereby, the concentration of the liquid concentrate solids is increased stepwise. For example with respect to whey, the concentration of the liquid concentrate from the first unit may be 66 %, and from the second unit 73 % starting with 58 % in the liquid product. For each unit recirculation of the liquid concentrate within the unit may take place as described above. The method is even more energy-efficient when it is carried out stepwise.

Furthermore, in one embodiment a secondary stream is fed to the stream of paste concentrate, such as described in WO 00/72692.

Turning to the figure, a preferred system comprising a concentration unit for concentrating is shown. The liquid product is fed to the system via a conduit (1) to a recirculation conduit (2). The recirculation conduit (2) leads to a heat exchanger (3), wherein the liquid product is heated to a temperature above the crystallisation temperature of the liquid product. The heat exchanger (3) is preferably a plate heat exchanger. The heat exchanger (3) is heated by steam or hot water which is led to the heat exchanger (3) through a conduit (15) and out through a conduit (16). Boiling

in the heat exchanger is suppressed by a control valve (4). The liquid product is transferred into a separator vessel (5). Flash separation is carried out in the vessel (5), whereby the vapour is removed via a conduit (14) and the liquid concentrate obtained is removed via a conduit (6). In the preferred embodiment, the liquid concentrate obtained in conduit (6) is divided into a main stream flowing in conduit (8) and a recirculation stream flowing in conduit (7). The stream in conduit (7) is mixed with liquid product from conduit (1) and recycled to the heat exchanger (3) through conduit (2).

Conduit (8) takes the concentrate to a (water) cooled vessel (18) via a pump (19) directly into an air dryer (17). The dryer is preferably a spray/fluid bed dryer.

Example

The feed material for the experiment was pre-treated rennet cheese whey with 6,00 % total solids and permeate from the production of milk protein by ultrafiltration of sweet whey with 5,50 % total solids.

In all Experiments whey or permeate was pre-concentrated in a multistage MVR falling film evaporator with 50 mm tubes of 18 m length as described by e.g. Knipschildt in Modern Dairy Technology (Ed. Robinson) 1986, Vol 1, p. 147 ff.

The experiments were performed according to the invention in a system as the one illustrated in the drawings. The concentrate from the falling film evaporator (not shown in the drawings) was fed to the recirculation line of a suppressed boiling forced circulation plate evaporator. After heating the separation process took place in a flash separator (a Single effect Paraflash, manufactured by APV Separation Processes). The concentrate from the exit line was then led to the cooler and subsequently fed directly to a spray dryer.

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	Feed kg/h	3300	3000
	TS in feed %	58	58
	TS in concentrate from evaporator %	83	76
35	Drying air inlet temperature C	175	180

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Drying air outlet temperature C
Residual moisture in powder %
Flowability (1)
Caking properties (1)

82	85
2	2
excellent	good
"moderate"	"moderate"

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(1) Referece is made to the text above

CLAIMS

1. A method for converting a liquid product into substantially free-flowing powder, said method comprising the steps of

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- heating the liquid product to a temperature above the crystallisation temperature of any component in the liquid product in a heat exchanger,
- flash separating volatile components from said heated liquid product to obtain a paste concentrate,
- 10 - pre-cooling a fraction of said paste concentrate, and
- drying said combination product.

2. The method according to claim 1, wherein the pre-cooling step is conducted by passing the paste concentrate through a cooled vessel having a vessel wall, whereby at most 50 % of the paste concentrate is in contact with the vessel wall during passing.

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3. The method according to claim 1 or 2, wherein at most 50 % of the paste concentrate is crystallised when entering the dryer.

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4. The method according to any one of the preceding claims, wherein the drying step is performed in a spray/fluid bed dryer to obtain a free-flowing powder.

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5. The method according to any one of the preceding claims, wherein the liquid product is selected from acid whey, sweet whey, salt or demineralised whey, lactose, mother liquor from production of lactose and permeate from production of protein from milk or whey.

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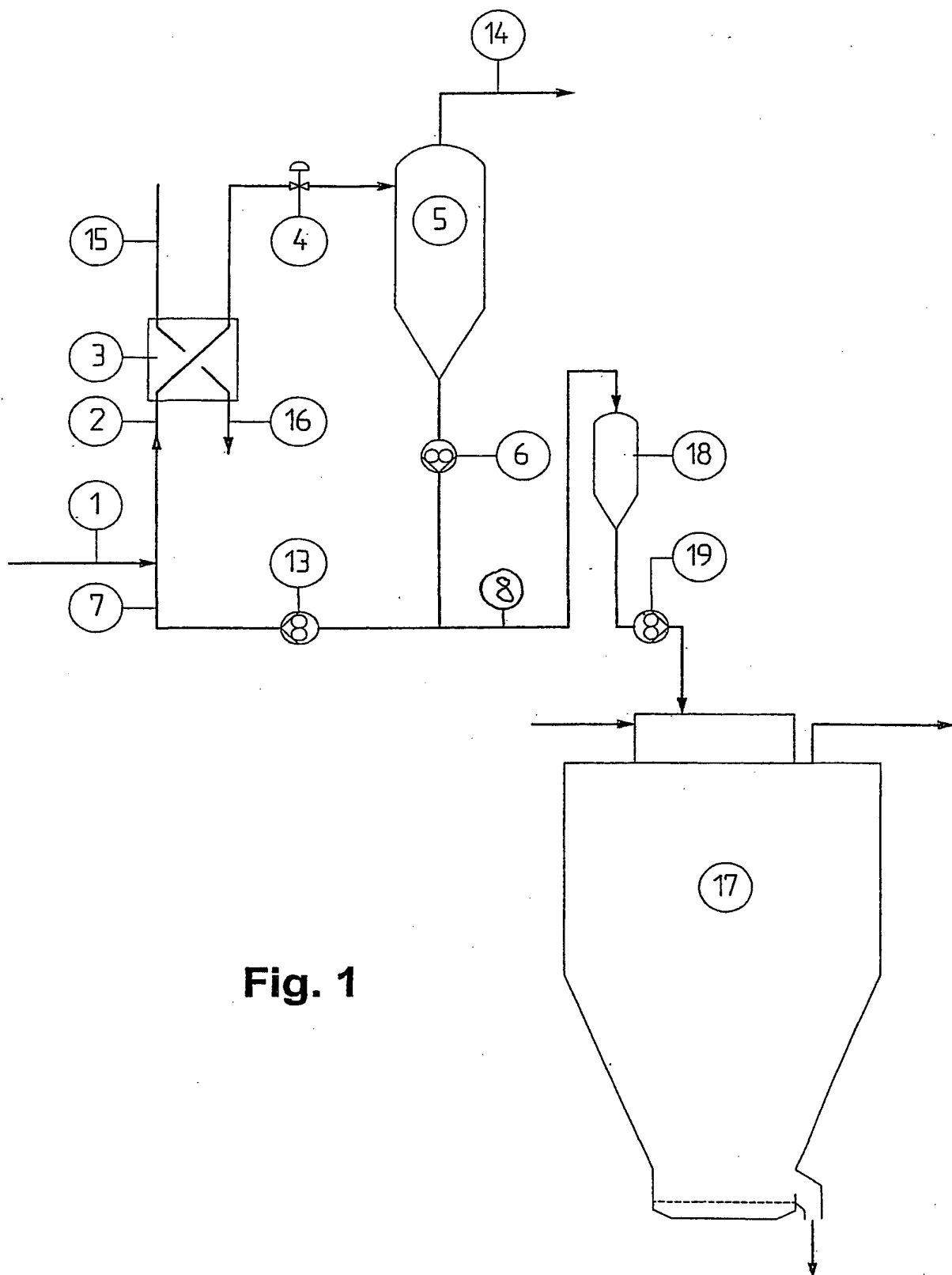
6. The method according to any one of the preceding claims, wherein the heat exchanger is a plate heat exchanger

7. The method according to any one of the preceding claims, wherein the liquid product comprises at least 50 % of solid, preferably at least 60 %, more preferably at least 70 % of solid.

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8. The method according to any one of the preceding claims, wherein the product temperature during the flash separation is in the range of 65-98°C, more preferably in the range of 70-96°C, even more preferably in the range of 80-96°C.
- 5 9. The method according to any one of the preceding claims, wherein at least 75 % of the paste concentrate obtained is mixed with liquid product and recycled to the heat exchanger to be reheated and subjected to the flash separating step.
- 10 10. The method according to any one of the preceding claims, wherein at most 25 % of said paste concentrate is pre-cooled.
- 15 11. A system for concentrating a liquid product comprising means (2) for feeding a heat exchanger, a heat exchanger (3) and a separator vessel (5), a cooled vessel (18) and a pump (9), and a dryer, connected by suitable conduits.
- 20 12. The system according to claim 11, wherein the heat exchanger (3) is a plate heat exchanger.
13. The system according to claim 11 or 12, wherein the separator vessel (5) is a flash separator.
14. The system according to any one of claims 11-13, further comprising a secondary conduit (7) extending in a closed loop connection from the separator vessel (5) to the heat exchanger (3).

1/1

**Fig. 1**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/DK 02/00271

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: A23C 21/00, A23C 1/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: A23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 0072692 A1 (APV ANHYDRO A/S), 7 December 2000 (07.12.00), abstract, page 3, line 32 - line 39; page 12, line 34 - line 37; claims 17-33 --	1-14
X	WO 8603942 A1 (APV ANHYDRO A/S), 17 July 1986 (17.07.86), abstract, page 3, line 2 - line 20; claim 1 --	1-14
X	WO 9735486 A1 (APV ANHYDRO A/S), 2 October 1997 (02.10.97), abstract, page 4, line 3 - line 20; claim 1 --	1-10

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents:

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- "P" document published prior to the international filing date but later than the priority date claimed

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/DK 02/00271

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3615663 A (JAMES JOSEPH BECKER), 26 October 1971 (26.10.71) -- -----	1-14

INTERNATIONAL SEARCH REPORT

Information on patent family members

06/07/02

International application No.

PCT/DK 02/00271

Patent document cited in search report			Publication date	Patent family member(s)		Publication date
WO	0072692	A1	07/12/00	AU	4911800 A	18/12/00
				EP	1187534 A	20/03/02
				US	6335045 B	01/01/02
WO	8603942	A1	17/07/86	AT	40507 T	15/02/89
				DE	3567998 D	00/00/00
				DK	164764 B,C	17/08/92
				DK	410086 A	28/08/86
				EP	0205601 A,B	30/12/86
				IE	57150 B	06/05/92
				NO	863360 A	20/08/86
WO	9735486	A1	02/10/97	AU	2287097 A	17/10/97
				EP	0896510 A	17/02/99
				US	6048565 A	11/04/00
US	3615663	A	26/10/71	NONE		